



## **Performance and Durability of Advanced Environmental Barrier Coating Systems**

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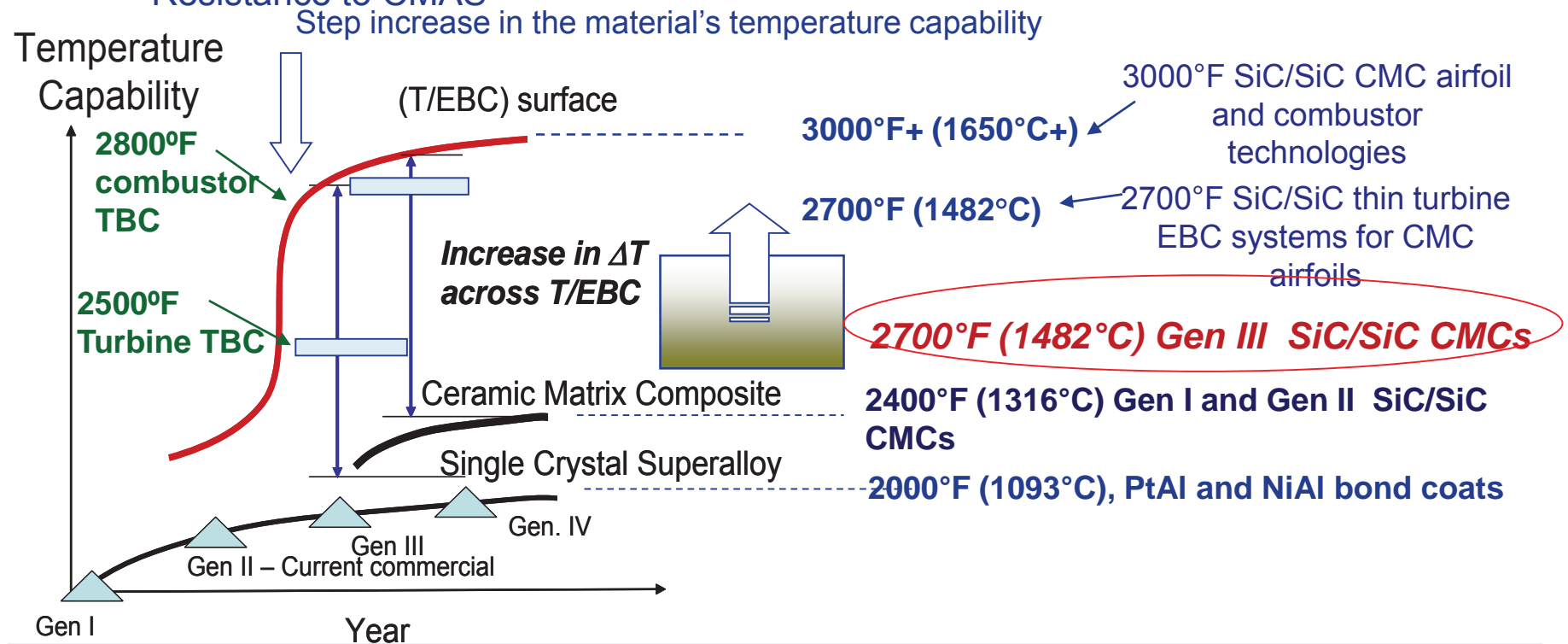


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## NASA Advanced EBC and CMC System Development

- **Emphasize temperature capability, performance and *long-term* durability**
  - Focus on highly loaded EBC-CMC Systems
  - 2700-3000°F (1482-1650°C) turbine airfoil and CMC combustor coatings
  - 2700°F (1482°C) EBC bond coat technology for supporting next generation turbine engines
    - Recession: <5 mg/cm<sup>2</sup> per 1000 h
    - Coating and component strength requirements: 15-30 ksi, or 100 - 207 Mpa
    - Resistance to CMAS





## Outline

- **Advanced environmental barrier coating (EBC) system development: Prime-reliant coating design as consideration**
- **Advanced bond coat developments, including HfO<sub>2</sub>-Si and Rare Earth-Si systems**
  - Recent developments on HfO<sub>2</sub>-Si based bond coat and multicomponent (Yb,Gd,Yb)<sub>2</sub>Si<sub>2-2x</sub>O<sub>7-x</sub> EBCs, integrated with 3D architecture CVI+PIP SiC/SiC ceramic matrix composites
    - Optimizing compositions and processing
    - Determining fundamental properties and upper use temperature limits
- **Durability considerations: advanced 2700°F+ capable EBC developments**
  - Focus on EBC-CMC system approaches, creep - fatigue – environmental interactions: rig durability demonstrations
  - Innovative modeling in supporting the coating developments, design tools, and life prediction
- **Environmental resistance, durability and component tests**
  - The EBC durability evaluations
  - Continuing the various rig tests, improving technology readiness levels, and transitioning EBCs for engine tests
- **Summary and conclusions**



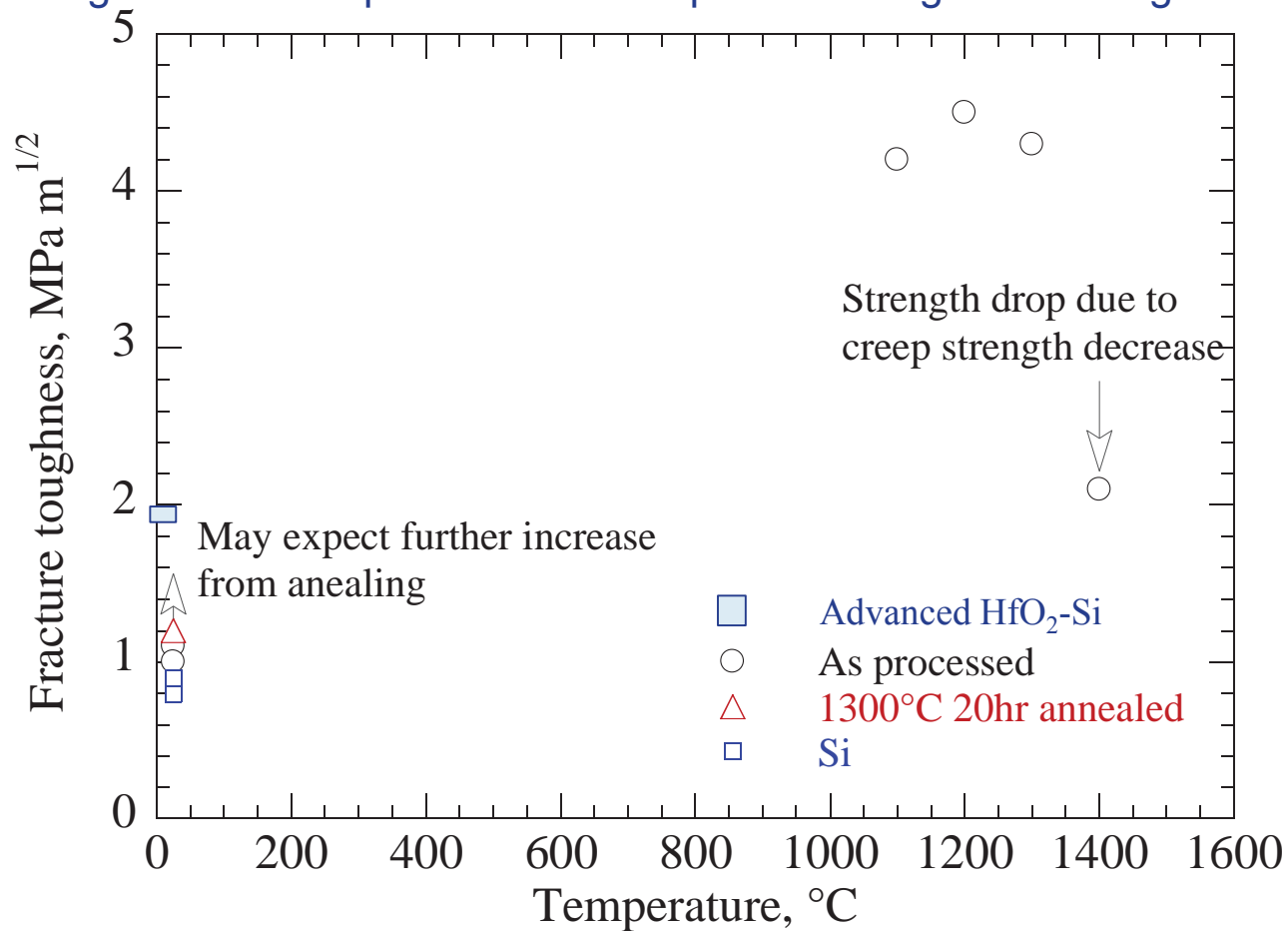
## NASA EBC and CMC System – Prime-Reliant Design Considerations

- Temperature capability is crucial for long-term durability, among other coating requirements, such as water vapor stability and phase durability, for advanced high pressure, high bypass turbine engines
  
- Advanced EBCs require high strength and toughness to be prime-reliant
  - Resistance to heat-flux (thermal gradients), high pressure combustion environment, creep-fatigue loading interactions
  - Bond coat cyclic oxidation resistance
  
- EBCs need erosion, impact and calcium-magnesium-alumino-silicate (CMAS) resistance and interface stability
  - Emphasize the multiple mechanism interactions
  
- EBC-CMC systems with affordable processing
  - Using existing infrastructure and alternative coating production processing systems, ensuring high stability coating systems, including Plasma Spray, EB-PVD and Directed Vapor EB-PVD, and/or emerging Plasma Spray - Physical Vapor Deposition
  - Affordable and safe, suitable for various engine components



## High Toughness HfO<sub>2</sub>-Si Bond Coat Composition Development

- HfO<sub>2</sub>-Si Bond coats showed high toughness
  - Toughness >4-5 MPa m<sup>1/2</sup> achieved
  - Emphasis on improving the lower temperature toughness, eliminating free Si or SiO<sub>2</sub>
  - Annealing effects on improved lower temperature toughness being studied

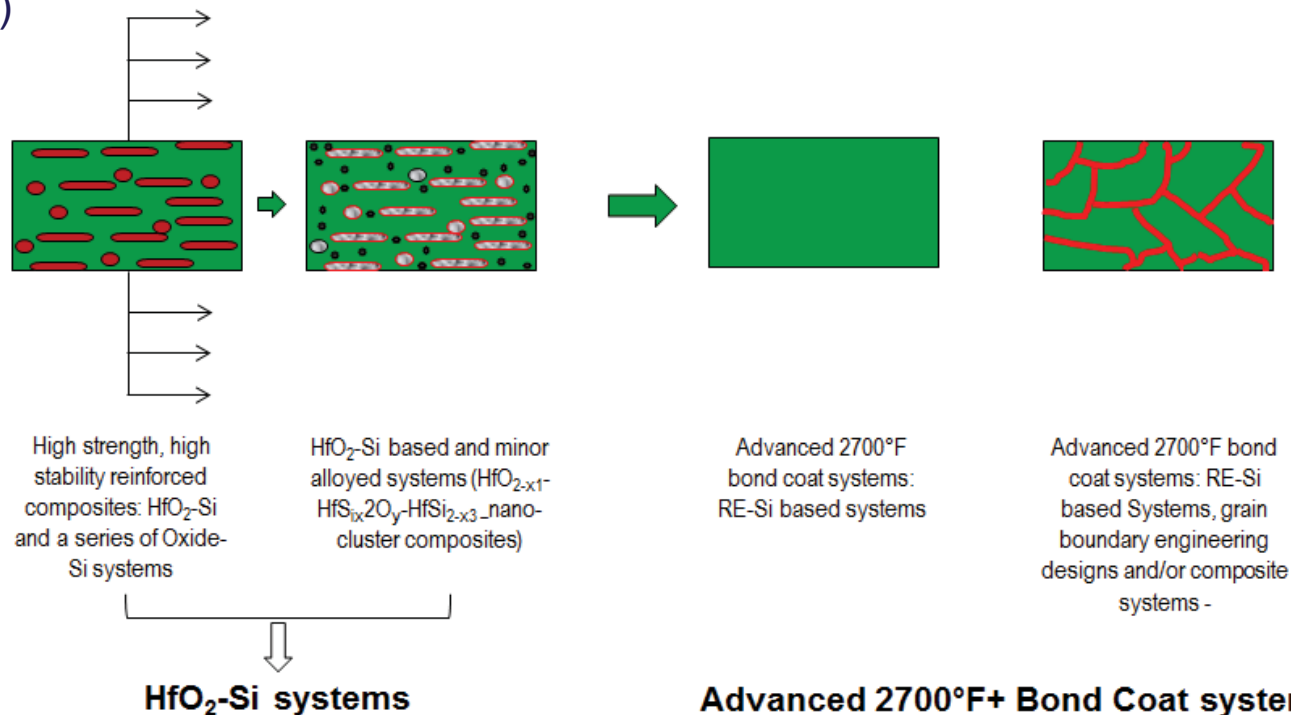




## NASA Advanced EBC - Bond Coat Systems

### NASA EBC Systems

- $\text{HfO}_2$  - $\text{RE}_2\text{O}_3$ - $\text{SiO}_2$ / $\text{RE}_2\text{Si}_{2-x}\text{O}_{7-2x}$  environmental barrier systems
  - Controlled silica content and rare earth dopant content to improve EBC stability, toughness, erosion and CMAS resistance
  - $\text{HfO}_2$ -Si based bond coat, controlled oxygen partial pressure via compositions
  - Advanced rare earth-Si composition systems for 2700°F+ long-term applications
- Early  $\text{RE}_2\text{O}_3$ - $\text{SiO}_2$ - $\text{Al}_2\text{O}_3$  or YAG Systems
- Develop prime-reliant composite EBC-CMCs, HfSiRE(CN) systems (beyond Hf-RE-Si based bond coats)

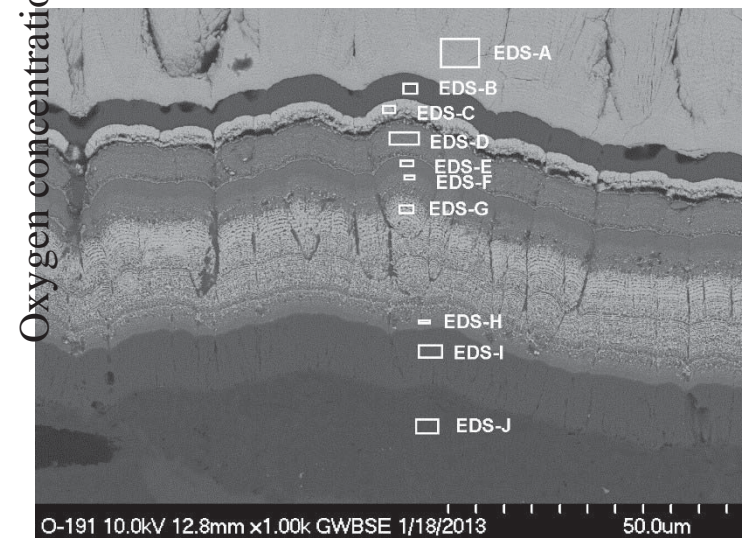
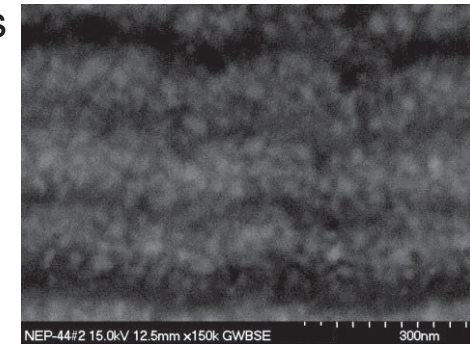
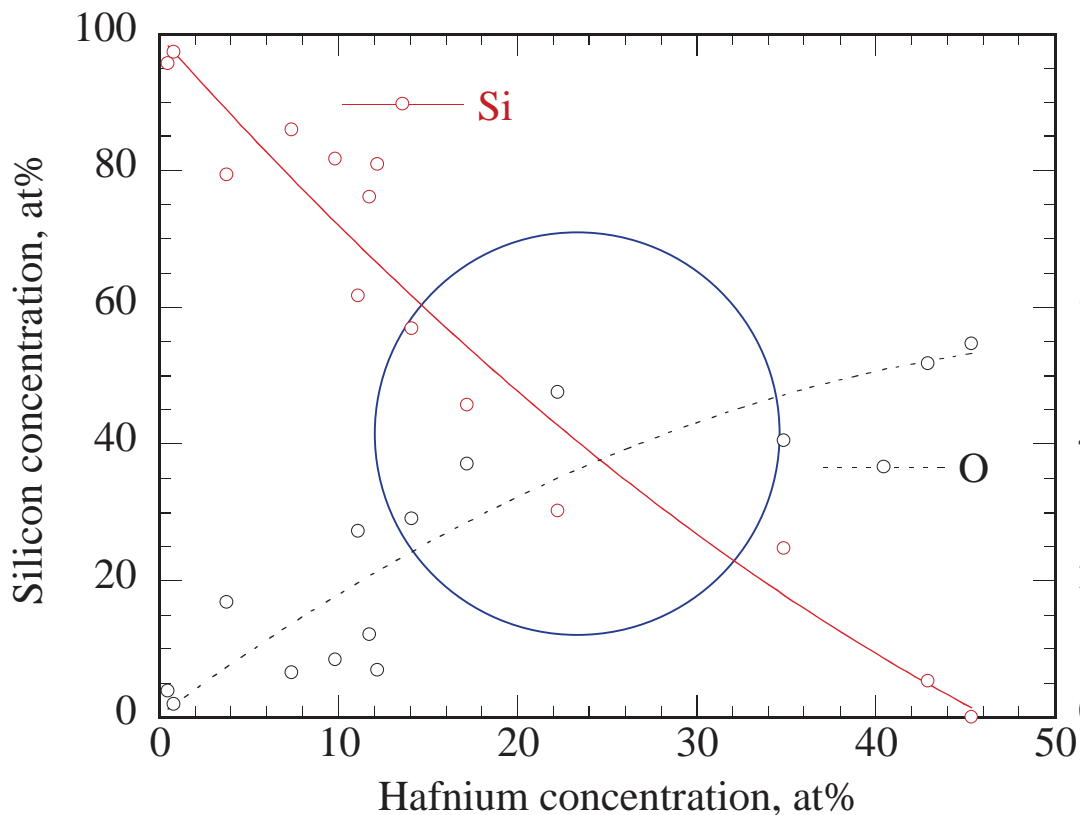


Bond coat systems for prime-reliant EBCs; capable of self-healing

## HfO<sub>2</sub>-Si Bond Coats EB-PVD Processing and Composition Optimizations



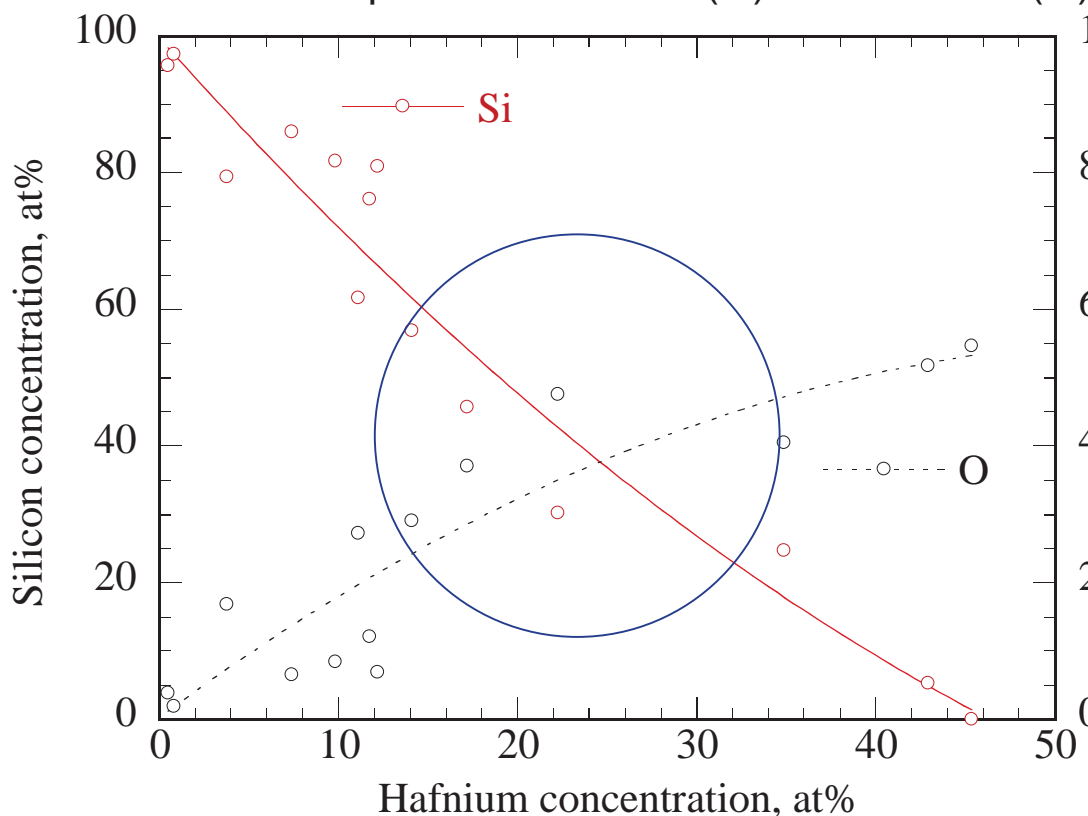
- Early EB-PVD HfO<sub>2</sub>-Si bond coat process and composition optimizations
- Achieving lower oxygen, low silicon, robust processing, and durable coatings at the SiC/SiC-bond coat interface
- Controlling pO<sub>2</sub> was a major objective
- Similar developments for RE-Si (O) and RE-Hf-Si(O) bond coats



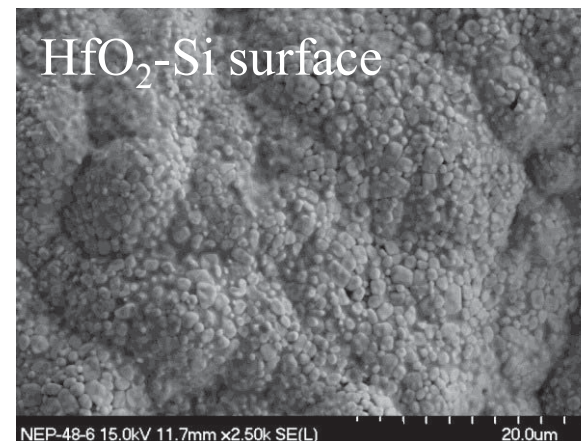
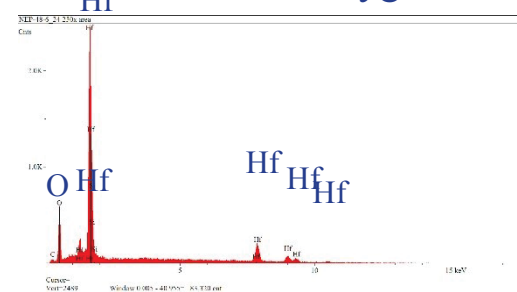
# HfO<sub>2</sub>-Si Bond Coats EB-PVD Processing and Composition Optimizations - Continued



- Early EB-PVD HfO<sub>2</sub>-Si bond coat process and composition optimizations
- Preferred HfO<sub>2</sub>, Si co-deposition, or hybrid HfO<sub>2</sub>, Si co-deposition + alternating layering structures
- Achieving lower oxygen, low silicon, robust processing, and durable coatings at the SiC/SiC-bond coat interface, controlling pO<sub>2</sub> was a major objective
- Similar developments for RE-Si (O) and RE-Hf-Si(O) bond coats



Low and controllable oxygen content



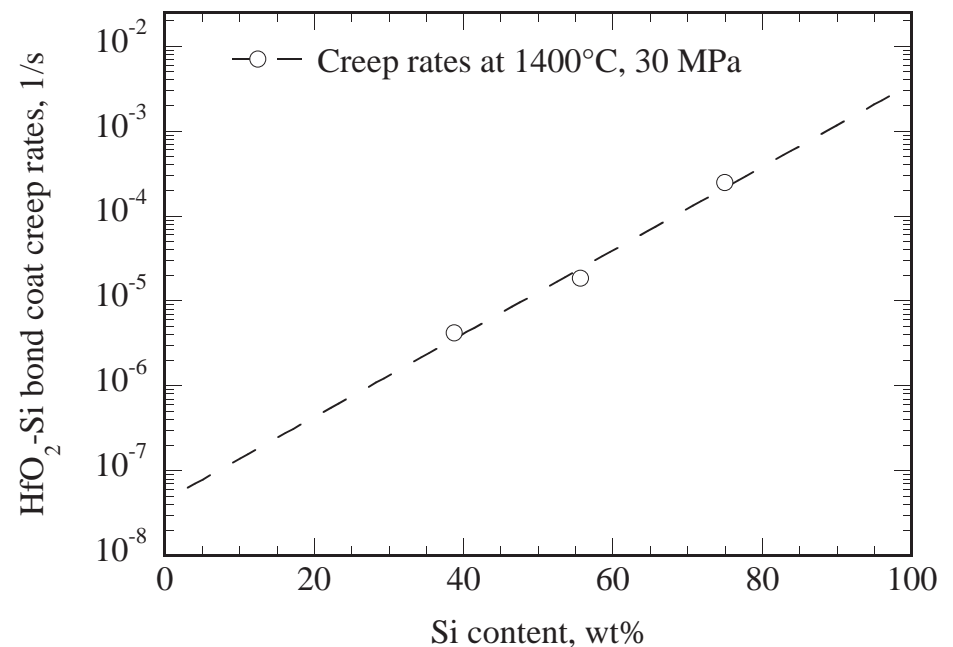
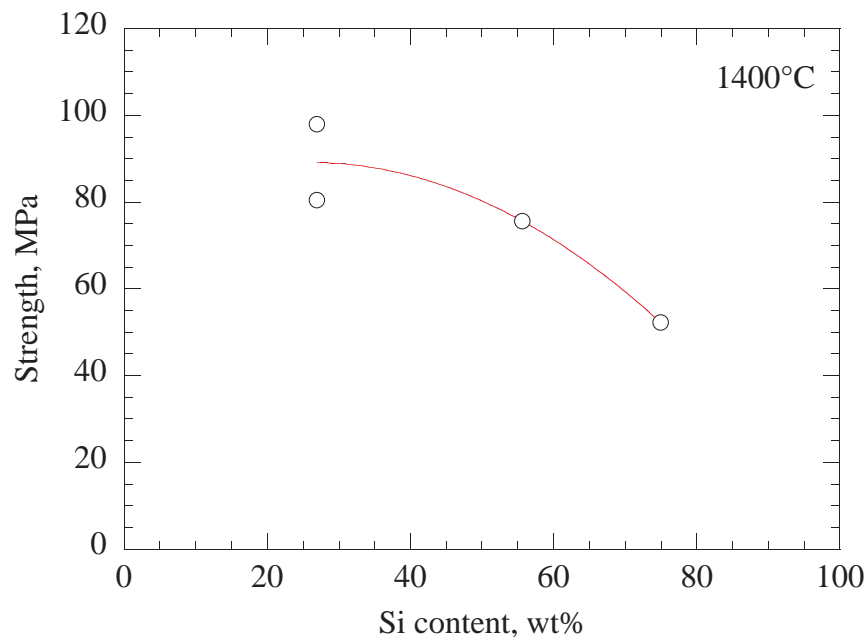
1500°C, 50h, laser heat flux rig tested





## Effects of Compositions on $\text{HfO}_2$ -Si Strength and Creep Rates

- The composites coatings have improved creep strength, and creep resistance at high temperatures
- *Increased  $\text{HfO}_2$ - $\text{HfSiO}_4$  contents improve high temperature strength and creep resistance*
- *Low diffusion with controlled oxygen content, and  $\text{HfO}_2$ - $\text{HfSi}_x\text{O}_y$*

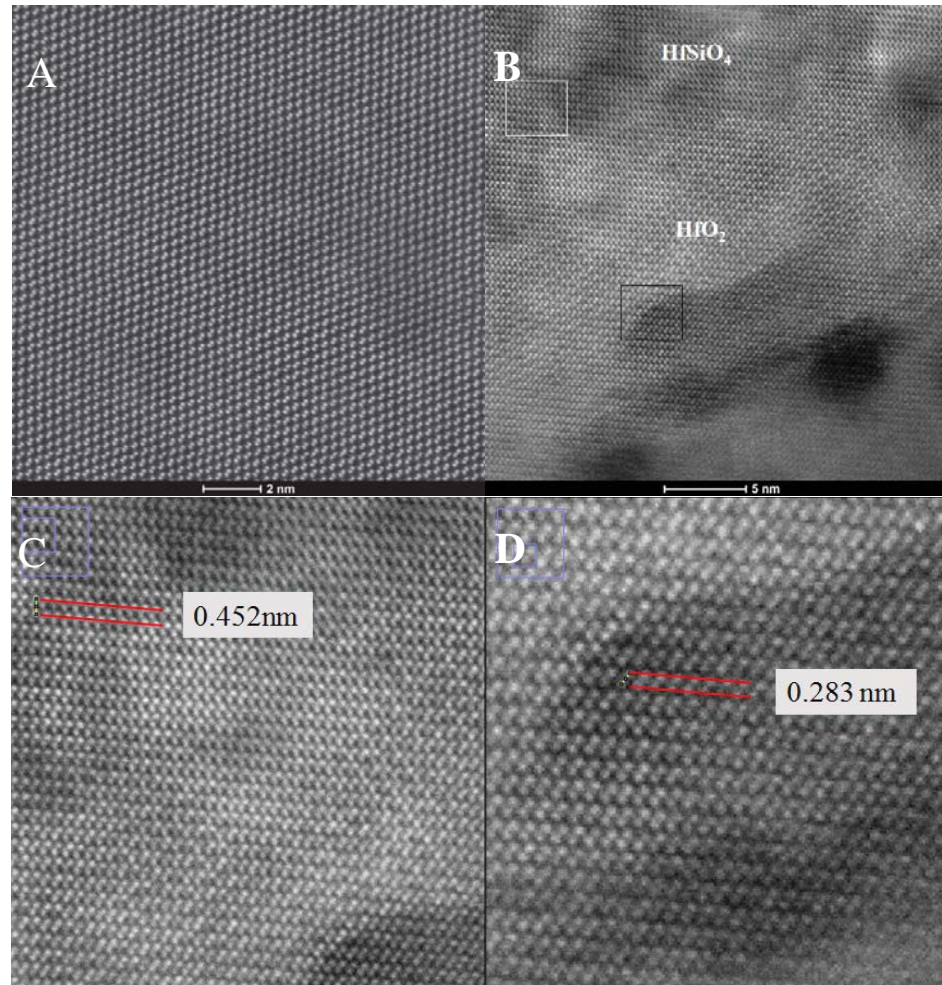


Early test results from processed  $\text{HfO}_2$ -Si bulk specimens (Zhu, ICMCTF 2014)



## Advanced 2700°F+ HfO<sub>2</sub>-Si Bond Coats

- High Resolution TEM Images showing advanced compositions ensuring high strength, high stability, high toughness, and low diffusion



HRTEM of Si matrix. B) HRTEM of HfO<sub>2</sub>-HfSiO<sub>4</sub> structure. C) Zoomed in view of HfSiO<sub>4</sub> structure in B) showing 4.52 Å spacing of (101) plane. D) Zoomed in view of HfO<sub>2</sub> structure in B) showing 2.83 Å spacing of (111) plane.

A. L. Robertson, F. Solá, D. Zhu, J. Salem, K. W. White, *Microscale Fracture Testing of HfO<sub>2</sub>-Si Environmental Barrier Coatings*, in press.



## Recent Testing and Development of NASA Advanced Multicomponent Yb-Gd-Y Silicate EBC/HfO<sub>2</sub>-Si System on 3D Architecture CVI+PIP SiC/SiC CMC under 2700°F+ SPLCF Conditions

- Two EBC specimens tested under the laser heat flux test rig under 10 ksi (500 hr) and 15 ksi (140 hr completed) SPLCF conditions, respectively, durability tested in air
- Advanced EBC-CMC specimens tested in isothermal furnace test at 2700°F, 300 h completed for comparisons
- Various laser tests for coating composition down-selects and failure mechanism modeling



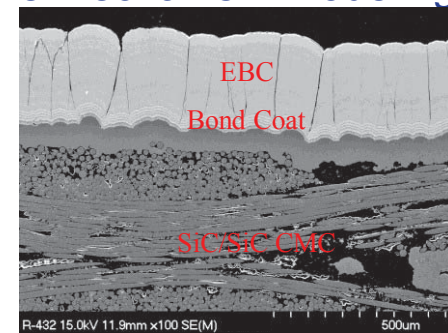
RB2014-54-4, EBC 512h/CMC 492h



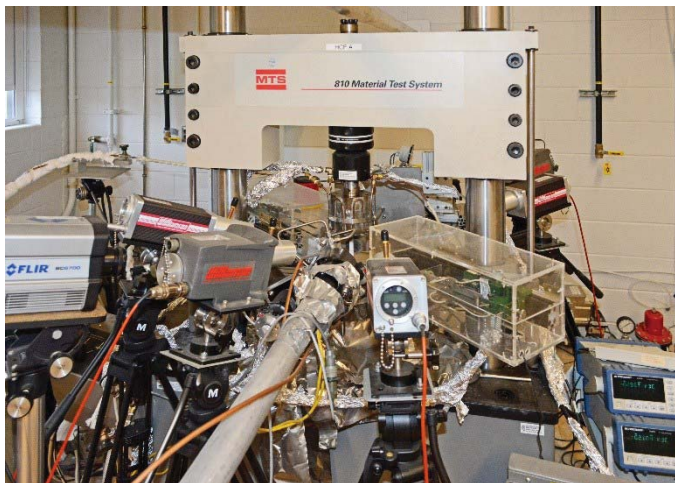
RB2014-54-6, EBC 140h



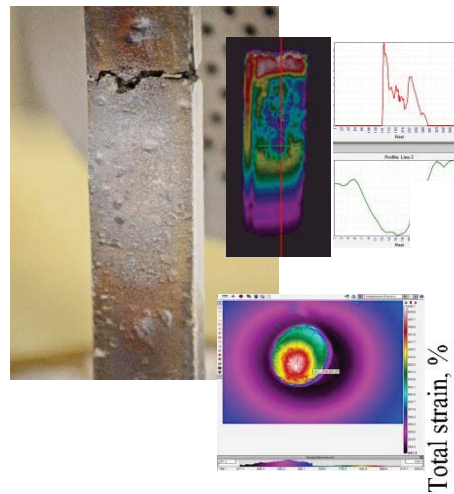
RB2014-54-8, Isothermal furnace 300hr



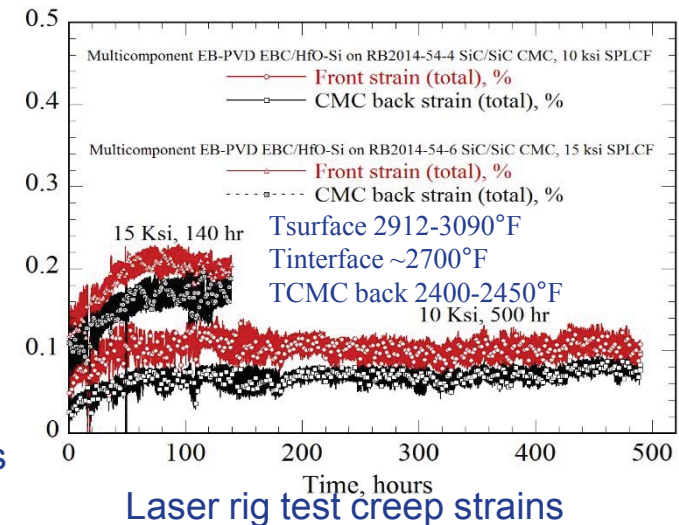
Example EBC cross-section



Laser III MTS 810 Test rig



Developing laser rig based NDE and in-plane thermal conductivity measurements





## Laser Rig Testing and Advanced EBC Development

- Multicomponent EBC vane process developments, for rig and component testing
- Witness specimens also processed for evaluation
- CMAS testing response under heat flux and furnace
- Laser steam tested HfO<sub>2</sub>-Si bond coat specimens



RB2014-54-4, EBC  
512h/CMC 492h



RB2014-54-6, EBC 140h



EBC 296



EBC 297

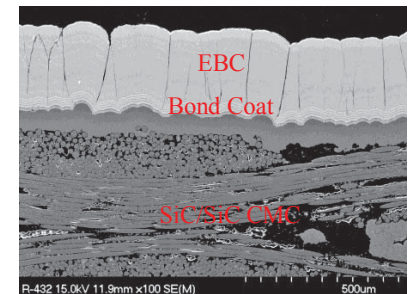


EBC 298

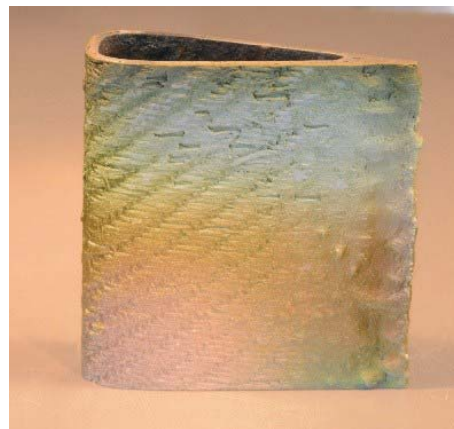


EBC 299

Witness Specimens Processed with EBCs (on 3D Architecture  
CVI+PIP CMCs)



Example EBC cross-section



Turbine vanes with EBCs

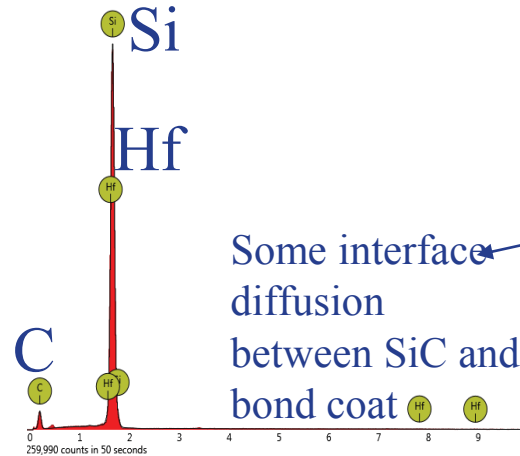
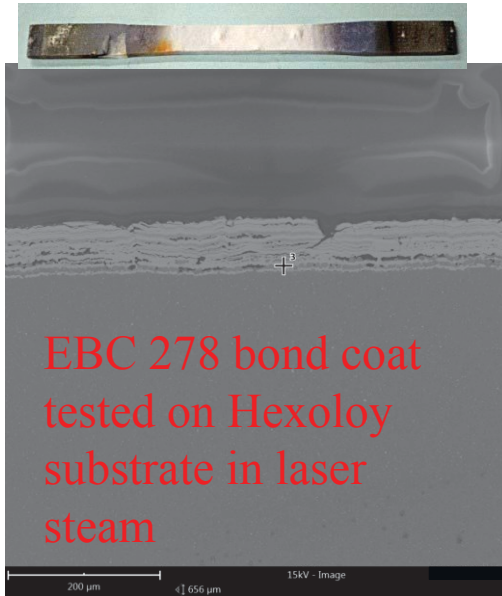


The TTT Augmentation Project  
Coated Turbine Vanes (Advanced EB-  
PVD NASA composition coatings)

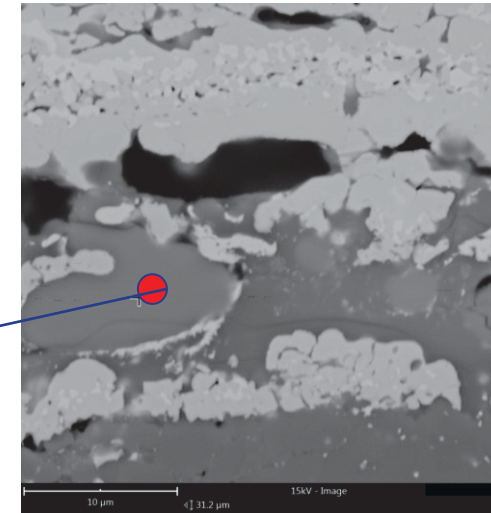
Laser rig test SPLCF creep strains



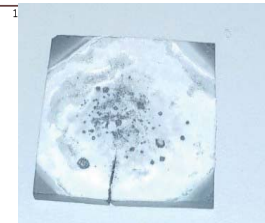
## Selected Recent Tested Specimens – EBC Tests



Some interface diffusion between SiC and bond coat



2700°F, 100 h laser steam cyclic test (1 h cycles), some interface diffusion and SiO<sub>2</sub> – rich phase separation (as we observed in the past)



278 HfO<sub>2</sub>-Si coating, Laser steam cyclic, 100hr



617: HfO<sub>2</sub>-Si



286



534



514

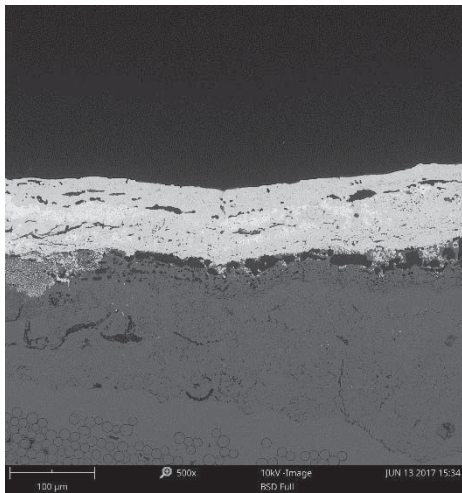
Selected steam furnace tested advanced HfO<sub>2</sub>-Si-EBC specimens early



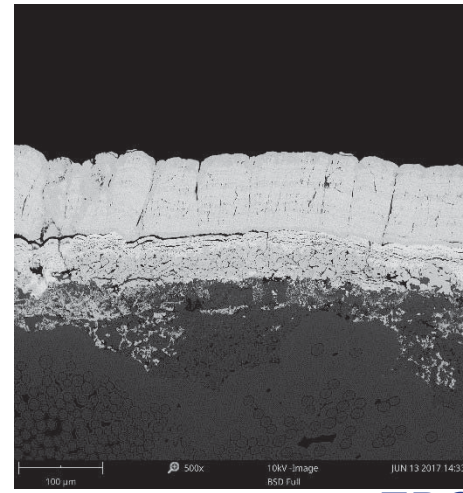
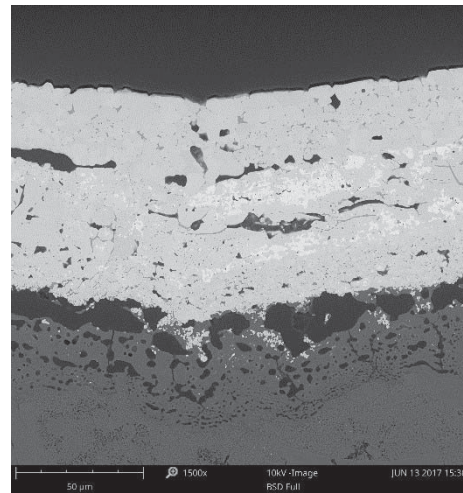
HfO<sub>2</sub>-Si, 50 h furnace cyclic, air



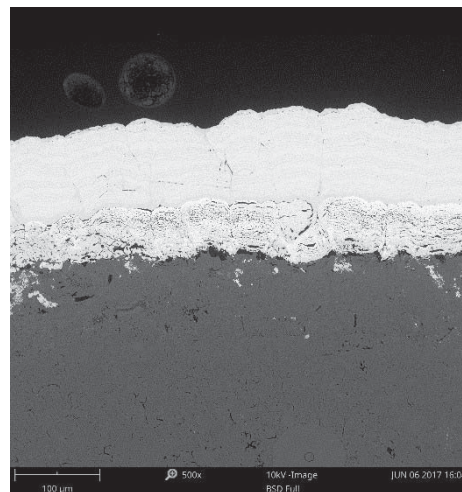
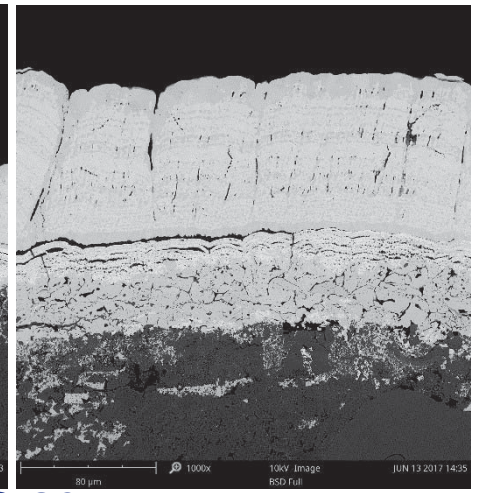
# Steam Cyclic Tests of Turbine Vane Turbine Vane Process Witness Samples – In a little more SiO<sub>2</sub> rich Steam Environments (2600°F on CVI+PIP CMC Substrates (Interface Reaction and Oxidation will be further Studied)



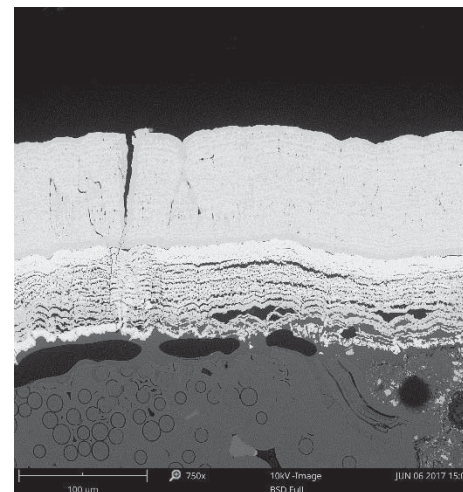
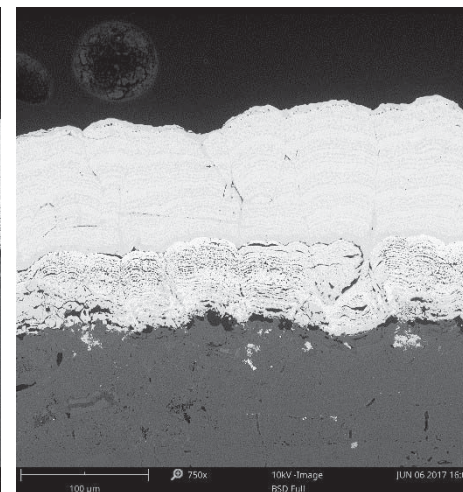
EBC 296 – had interface debond



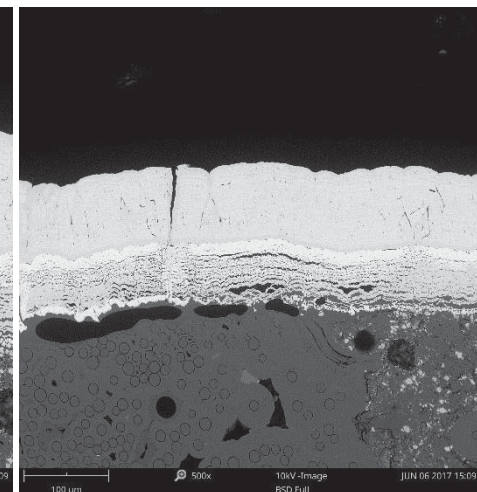
EBC 297



EBC 297



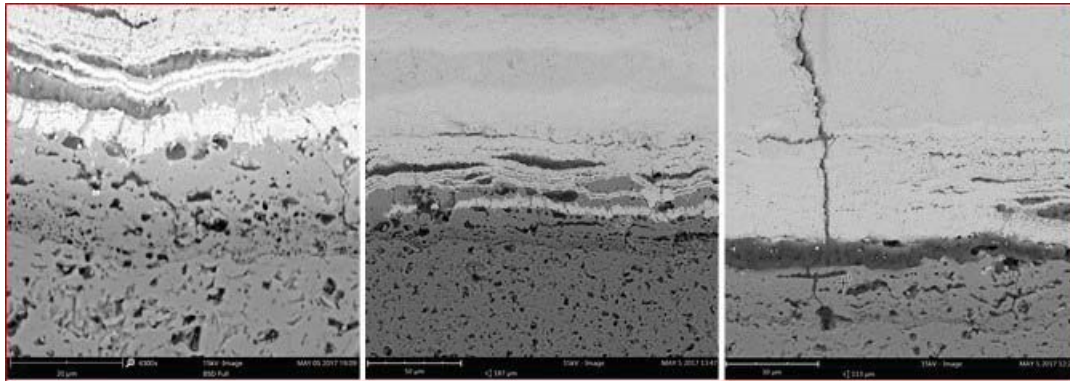
EBC 298





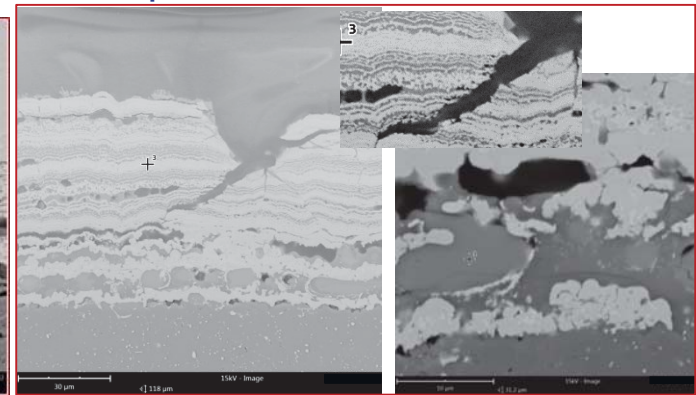
## Advanced EBC Development and Laser – High Heat Flux Rig Test Developments, understanding the Delamination Mechanics

- The work has been focused on the HfO<sub>2</sub>-Si bond coat composition effects and the diffusion barrier performance of HfO<sub>2</sub>-Si bond coats and NASA multicomponent EBCs.



HfO<sub>2</sub>-Si bond coat, interface reactions, SiO<sub>2</sub> formation in presence vertical cracks reactions

Furnace steam test (EBC 286 series), 1426°C (2600°F), 100 hr, observed porosity formation, SiO<sub>2</sub> rich phase separation from Bond coat, and SiO<sub>2</sub> formation from a vertical crack



HfO<sub>2</sub>-Si bond coat, heat flux delamination, some volatility of SiO<sub>2</sub> rich compositions, and interface reactions – high toughness bond coat is crucial  
Laser steam cyclic (EBC 278 series), 1500°C 100h

- Diffusion couples are being studied in understanding HfO<sub>2</sub>-Si bond coat diffusion and kinetics
- Expanding to SiHf-CN and HfSiRE-CN based high strength high toughness coating and/or CMC integration, and focusing on high-heat-flux test & stress tolerance

$$G = \sigma^2 h / 2\bar{E}$$

$$= [Eh(1+\nu)/(1-\nu)](\Delta\alpha\Delta T)^2 / 2$$

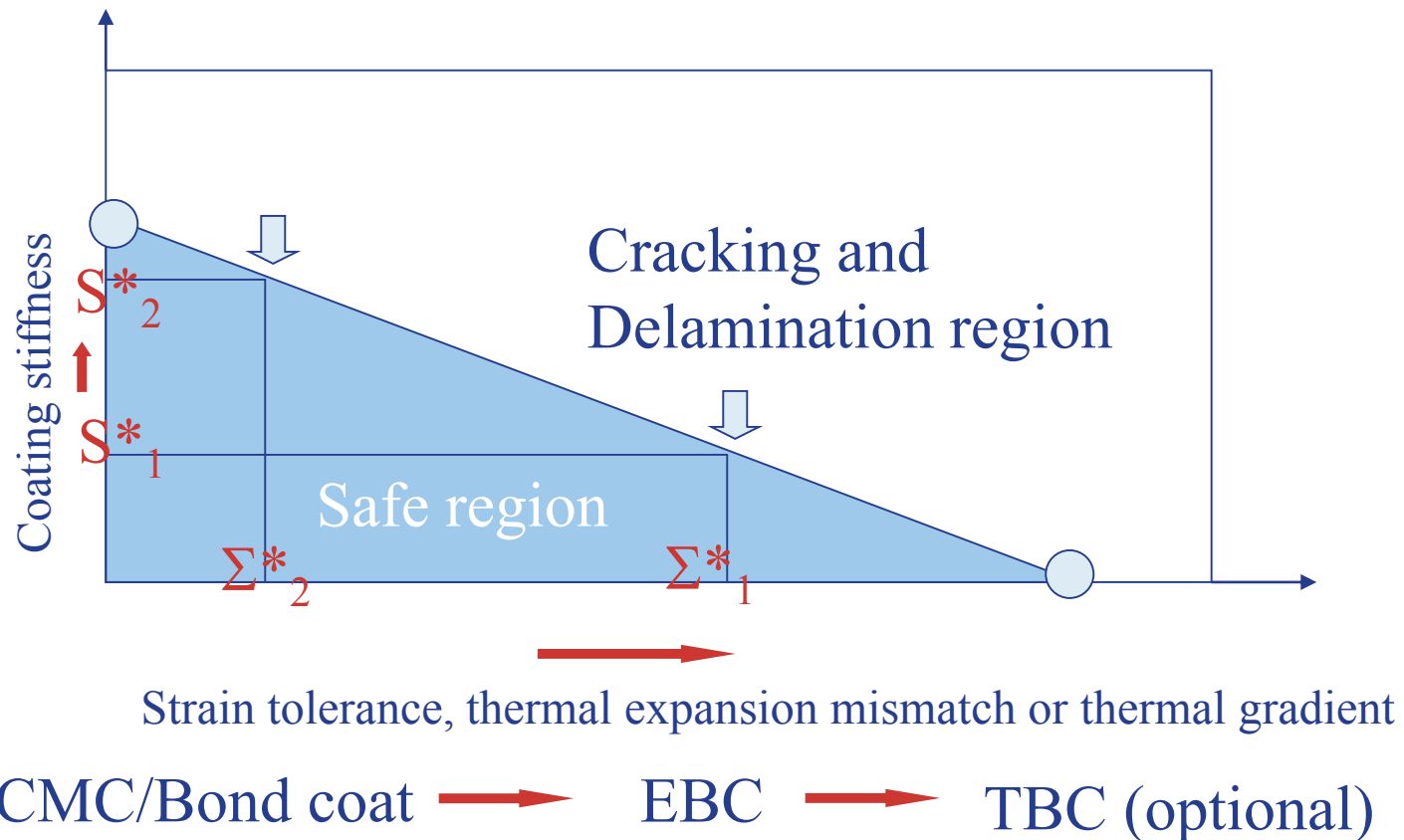
Modulus E has a strong effect on delamination driving force G



Laser high heat flux test rig



## Coating Safe Design Approach

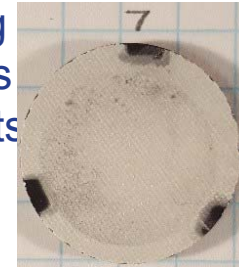




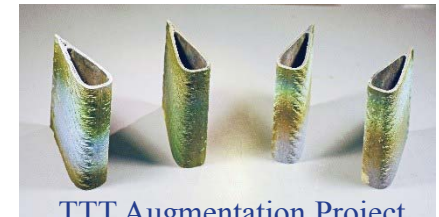


## Advanced EBC Development and Laser and JETS High Heat Flux Rig Test Development for Comparisons

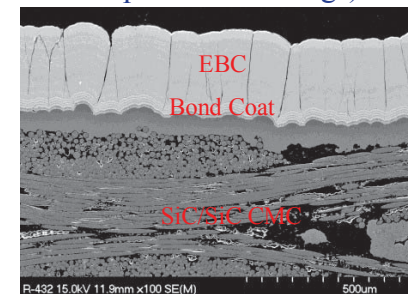
- Selected samples including the turbine vane samples being tested in high heat flux JETS rig (including the vane witness samples) in Praxair under a NASA contract, up to 100h tests including CMAS tests
- Turbine vane witness samples evaluated in the JETS tests
- Currently emphasis focused on comparisons of steam furnace, laser heat flux steam, and JETS tests
  - Crucial in studying advanced modeling and mechanism interactions



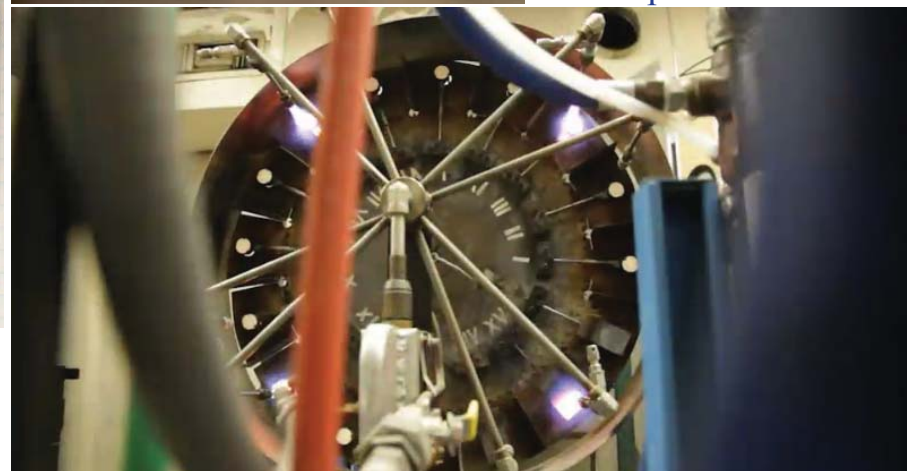
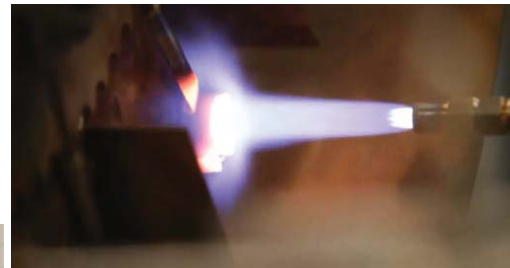
Witness samples  
Tested in JETS



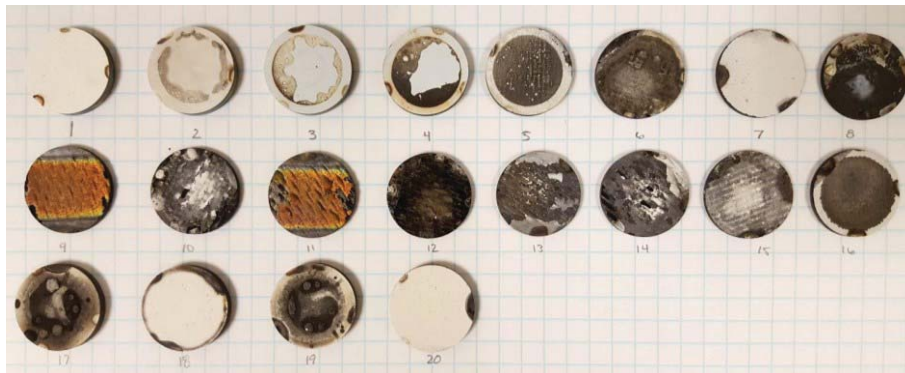
TTT Augmentation Project  
Coated Turbine Vanes  
(Advanced EB-PVD NASA  
composition coatings)



Example EBC cross-section



High heat flux JETS testing

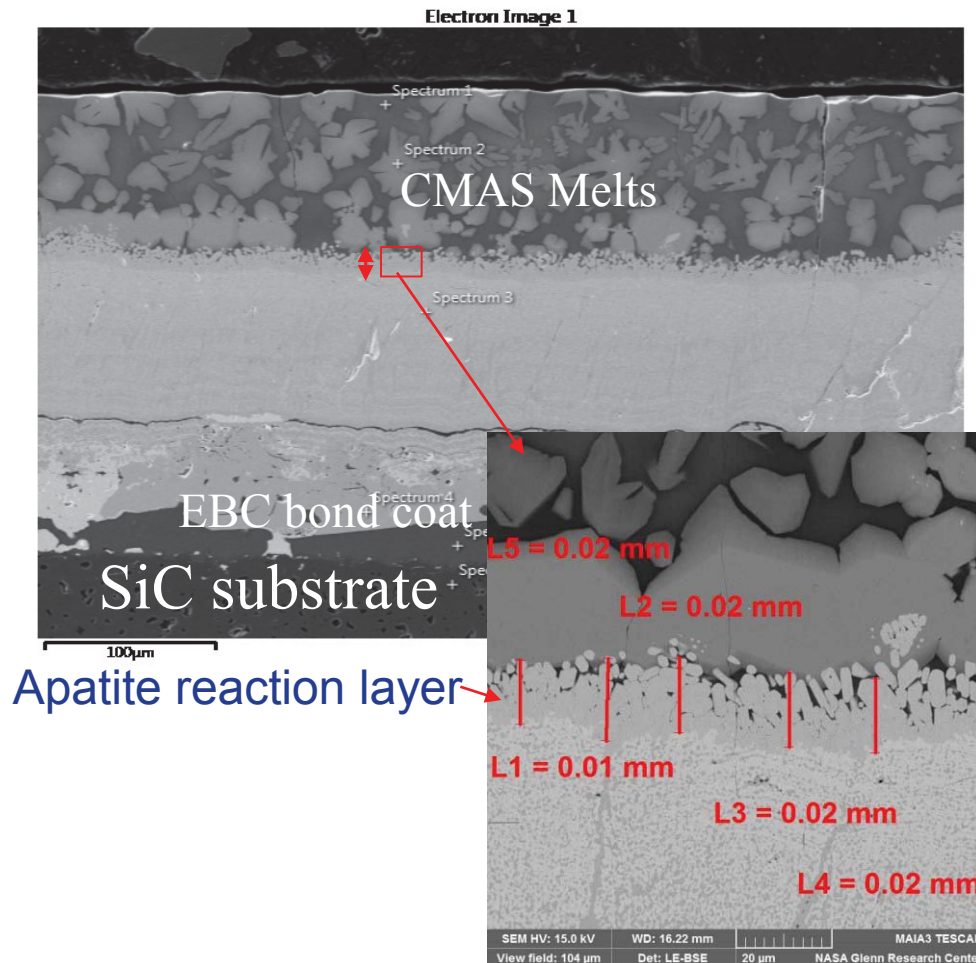


Some tested specimens

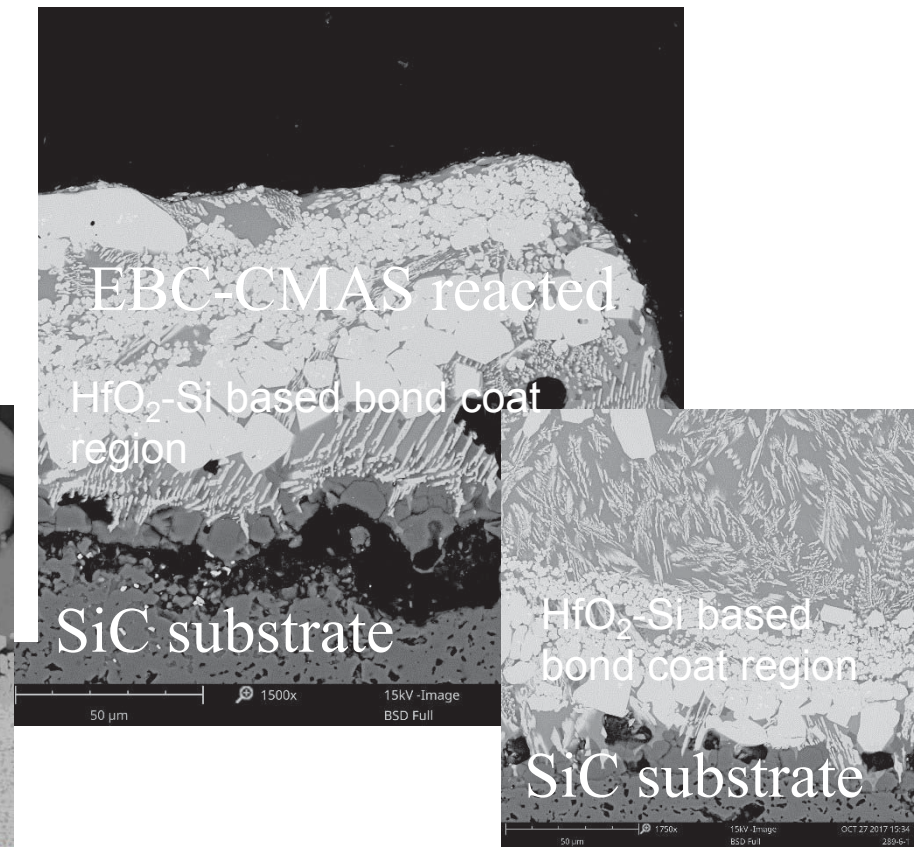


## Some CMAS Reaction Perspectives of NASA Multicomponent EBCs – Initial Test Results

- CMAS is of serious concern for EBCs
- Increasing coating temperature capability and reducing diffusion with defect cluster coating concepts are among the main approaches for improving CMAS resistance



1300°C, 5 h, furnace exposed, in air



1500°C, 100 hr, furnace exposed in air



## Summary and Conclusions

- Advanced HfO<sub>2</sub>-Si and Rare Earth- Silicon based bond coat compositions developed, composition and processing are still being optimized
- The coating has showed excellent oxidation resistance and protection for CMCs
- HfO<sub>2</sub>-Si EBC bond coat showed excellent strength, fracture toughness and thermal mechanical fatigue resistance
- Laser heat flux steam tests have been conducted and compared furnace steam cyclic tests, interface reactions will be further studied
- The coatings showed 2700°F operating temperature viability and initial durability on SiC/SiC ceramic matrix composites; continued processing optimization and robustness are being addressed
- The current emphasis has been placed on integration with CVI-PIP substrates, and also improving the CMAS resistance of advanced EBCs

### Future plans

- More advanced hafnium-rare earth silicate EBC-hafnium rare earth-Si (O) bond coat systems will be further investigated
- NASA advanced EBCs also included HfSiRECN systems for helping develop prime-reliant EBCs



## Acknowledgements

The work was supported by NASA Aeronautics Programs, and Transformational Tools and Technologies Project.